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HIGH-STRENGTH SPUTTERING TARGETS AND METHOD OF MAKING SAME

5   CROSS REFERENCE TO RELATED APPLICATIONS

This application is related to Application No. 09/098,761, filed June 17, 1998.

BACKGROUND OF THE INVENTION

10   The invention relates to sputtering targets and methods of making same; and to sputtering targets of high purity metals and alloys. Among these metals are Al, Ti, Cu, Ta, Ni, Mo, Au, Ag, Pt and alloys thereof, including alloys with these and or other elements. Sputtering targets may be used in electronics

15   and semiconductor industries for deposition of thin films. To provide high resolution of thin films, uniform and step coverages, effective sputtering rate and other requirements, targets should have homogenous composition, fine and uniform structure, controllable texture and be free from precipitates,

20   particles and other inclusions. Also, they should have high strength and simple recycling. Therefore, significant improvements are desired in the metallurgy of targets especially of large size targets.

A special deformation technique known as equal channel

25   angular extrusion (ECAE) described in U.S. Patents Nos. 5,400,633; 5,513,512; 5,600,989; and Patent No. 5,590,389 is used with advantage in accordance with the invention. The disclosures of the aforementioned patents are expressly incorporated herein by reference.

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SUMMARY OF THE INVENTION

The invention relates to a sputtering target made by a process including casting. The target has a target surface such that the surface of the target subjected to sputtering (referred

35   to as target surface) has a substantially homogeneous composition

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at any location, substantial absence of pores, voids, inclusions
and other casting defects, grain size less than about 1 μm and
5   substantially uniform structure and texture at any location.
Preferably, the target comprises at least one of Al, Ti, Cu, Ta,
Ni, Mo, Au, Ag, Pt and alloys thereof.

The invention also relates to a method of manufacturing a
target, as described above. The method comprises fabricating an
10   article suitable for use as a sputtering target comprising the
steps of:

    a.   providing a cast ingot;

    b.   homogenizing said ingot at time and temperature
sufficient for redistribution of macrosegregations and
15   microsegregations; and

    c.   subjecting said ingot to equal channel angular
extrusion to refine grains therein.

More particularly, a method of making a sputtering target
comprising the steps of:

20   a.   providing a cast ingot with a length-to-diameter
ratio up to 2;

    b.   hot forging said ingot with reductions and to a
thickness sufficient for healing and full elimination of case
defects;

25   c.   subjecting said hot forged product to equal
channel extrusion; and

    d.   manufacturing into a sputtering target.

Still more particularly, a method of fabricating an article
suitable for use as a sputtering target comprising the steps of:

30   a.   providing a cast ingot;

    b.   solutionizing heat treating said cast ingot at
temperature and time necessary to dissolve all
precipitates and particle bearing phases; and

    c.   Equal channel angular extruding at temperature
35   below aging temperatures.

1   33507/VGG/J104

        After fabricating as described to produce an article,
it may be manufactured into a sputtering target.

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1   33507/VGG/J104

DESCRIPTION OF THE DRAWINGS

　　　FIGS. 1A-1D are schematic diagrams showing processing steps
5　　of billet preparation for ECAE;

　　　FIG. 2 is a graph showing the effect of annealing
temperature on billet strength after 4 and 6 passes of ECAE for
Al 0.5 wt.% Cu alloy;

　　　FIG. 3A is a schematic diagram disclosing an apparatus for
10　gradient annealing of targets;

　　　FIG. 3B is a schematic diagram showing temperature
distribution through target cross-section C-C during gradient
annealing;

　　　FIG. 4 is an illustration of (200) pole figures for Al 0.5
15　wt.% Cu alloys processed with 2, 4 and 8 passes of route D, (in
FIG. 5) respectively;

　　　FIG. 5 is a graph showing the effect of number of passes and
route on texture intensity after ECAE of Al with 0.5 wt.% Cu;

　　　FIG. 6 is a graph showing the effects of annealing
20　temperature for route A after ECAE of Al with 0.5 wt.% Cu;

　　　FIG. 7 is a graph showing the effects of annealing
temperature on texture intensity for route B after ECAE of Al
with 0.5 wt.% Cu;

　　　FIG. 8 is a graph showing the effects of annealing
25　temperature on texture intensity for route C after ECAE of Al
with 0.5 wt.% Cu;

　　　FIG. 9 is a graph showing the effects of annealing
temperature on texture intensity for route D after ECAE of Al
with 0.5 wt.% Cu:

30　　　FIG. 10 is a pole figure illustrating the texture as a
result of the process described; and

　　　FIGS. 11, 11A and 11B are schematic diagrams of an apparatus
for ECAE of billets for targets.

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DETAILED DESCRIPTION

The invention contemplates a sputtering target having the following characteristics:

- substantially homogenous material composition at any location;
- substantial absence of pores, voids, inclusions and other casting defects;
- substantial absence of precipitates;
- grain size less than about 1μm;
- fine stable structure for sputtering applications;
- substantially uniform structure and texture at any location;
- high strength targets without a backing plate;
- controllable textures from strong to middle, weak and close to random;
- controllable combination of grain size and texture;
- large monolithic target size;
- prolonged sputtering target life;
- optimal gradient of structures through target thickness.

Targets possessing these characteristics are producible by the processes described.

Because of high purity, cast ingot metallurgy is useful in most cases for billet fabrication in target production. However, casting results in a very course dendritic structure with strong non-uniformity in the distribution of constitutive elements and additions across the ingot and large crystallites. Moreover, high temperature and long-time homogenizing cannot be applied in current processing methods because of the further increase of grains. One embodiment of the invention solves this problem by using homogenizing time and temperature sufficient for redistribution of macrosegregations and microsegregations followed by equal channel angular extrusion (ECAE) with a

sufficient number of passes, preferably from 4 to 6, for grain refinement.

Another embodiment eliminates other casting defects such as voids, porosity, cavities and inclusions which cannot be optimally removed by homogenizing and employs a hot forging operation.   In currently known methods hot forging has a restricted application because reductions are limited and are typically used at low temperature working for grain refinement. Other processes do not solve that problem when slab ingots of the same thickness as the billet for ECAE are used.   In the present invention, the as-cast ingot has a large length-to-diameter ratio, preferably up to 2.   During hot forging, the ingot thickness changes to the thickness of the billet for ECAE.   That provides large reductions which are sufficient for full healing and elimination of cast defects.

Still another embodiment of the invention is directed to precipitate- and particle-free targets. With currently known methods precipitate-free material may be prepared by solutionizing at the last processing step. However, in this case heating to solutionizing temperatures produces very large grains. The present invention provides a method for fabricating precipitate-free and ultra-fine grained targets.   According to this embodiment of the invention, solutionizing is performed at a temperature and time necessary to dissolve all precipitates and particle bearing phases and is followed by quenching immediately before ECAE.   Subsequent ECAE and annealing are performed at temperatures below aging temperatures for corresponding material conditions.

A further embodiment of the invention is a special sequence of homogenizing, forging and solutionizing operations. As-cast ingots are heated and soaked at the temperature and for the length of time necessary for homogenizing, then cooled to the starting forging temperature, then forged to the final thickness

at the final forging temperature (which is above the
solutionizing temperature) and quenched from this temperature.
5   By this embodiment all processing steps are performed with one
heating. This embodiment also includes another combination of
processing steps without homogenizing: forging at a temperature
of about the solutionizing temperature and quenching immediately
after forging.

10   It is also possible in accordance with the invention to
conduct aging after solutionizing at the temperature and for the
length of time necessary to produce fine precipitates with an
average diameter of less than 0.5 μm. These precipitates will
promote the development of fine and uniform grains during
15   following steps of ECAE

An additional embodiment of the invention is a billet for
ECAE after forging. An as-cast cylindrical ingot of diameter d_o
and length h_o (FIG. 1A) is forged into a disk of diameter D and
thickness H (FIG. 1B). The thickness H corresponds to the
20   thickness of the billet for ECAE. Then two segments are removed
from two opposite sides of the forged billet such as by machining
or sawing (FIG. 1C), to provide a dimension A corresponding to
a square billet for ECAE (FIG. 1D). ECAE is performed in
direction "C" shown on FIG. 1C. After the first pass the billet
25   has a near-square shape if the dimensions of the ECAE billet
(AxAxH), the dimensions of the forged disk (DxH) and the
dimensions of the cast ingot $(d_o x h_o)$ are related by the following
formulae:

$$D=1.18A$$

$$d_o{}^2 h_o = 1.39.A^2 H$$

30   The invention further contemplates the fabrication of
targets with fine and uniform grain structure. ECAE is performed
at a temperature below the temperature of static
recrystallization with the number of passes and processing route
35   adjusted to provide dynamic recrystallization during ECAE.

Processing temperature and speed are, correspondingly, sufficiently high and sufficiently low to provide macro- and
5   micro-uniform plastic flow.

A method for fabricating fine and stable grain structures for sputtering applications and to provide high strength targets is also provided. The billet after ECAE with dynamically recrystallized sub-micron structure is additionally annealed at
10   the temperature which is equal to the temperature of the target surface during steady sputtering. Therefore, the temperature of the target cannot exceed this sputtering temperature and for structure to remain stable during target life. That structure is the finest presently possible stable structure and provides
15   the best target performance. It also provides a high strength target. FIG. 2 shows the effect of the annealing temperature on the ultimate tensile strength and yield stress of Al 0.5 wt.% Cu alloy after ECAE at room temperature with 6 or 4 passes. In both cases as-processed material has high strength not attainable for
20   that material with known methods. Yield stress is only slightly lower than ultimate tensile strength. The increase of the annealing temperature in a range from 125°C to 175°C that, it is believed, corresponds to possible variations of sputtering temperature results in the gradual decrease of strength.
25   However, even in the worst case with an annealing temperature of 175°C target strength and, especially, yield stress are much higher than the strength of aluminum alloy AA6061 at T-O condition which is the most widely used for fabrication of backing plates (see FIG. 2). Thus, among other things, the
30   invention provides the following significant advantages:
- High strength monolithic targets may be fabricated from mild materials like pure aluminum, copper, gold, platinum, nickel, titanium and their alloys.

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    –   It is not necessary to use backing plates with
          additional and complicated operations such as
5   diffusion bonding or soldering.

    –   Fabrication of large targets is not a problem.

    –   Targets may easily be recycled after their sputtering
          life ends.

It is also useful to employ gradient annealing of targets

10 after ECAE. For that purpose a preliminary machined target is
exposed to the same thermal conditions as under sputtering
conditions and kept at those conditions a sufficient time for
annealing. FIG. 3 describes that processing. The target 1 is
fixed in a device 2 which simulates sputtering: a bottom surface

15 A of the target is cooled by water while a top surface B is
heated to the sputtering temperature. Heating is advantageously
developed at the thin surface layer by radiant energy q (left
side of FIG. 3A) or inductor 3 (right side of FIG. 3A) In
addition it is also possible to achieve gradient annealing of

20 targets directly in a sputtering machine at the regular
sputtering conditions before starting the production run. In all
these cases distribution of temperature through the target as
shown in FIG. 3B through sections C-C of FIG. 1 is non-uniform
and annealing takes place only inside a very thin surface layer

25 (δ). Following sputtering the same distribution is maintained
automatically. Thus, structural stability and high strength of
as-processed material are conserved for the main part of the
target.

An additional embodiment comprises a two-step ECAE

30 processing. At the first step ECAE is performed with a low
number of passes, preferably from 1 to 3, in different
directions. Then, the preliminary processed billet receives
aging annealing at low enough temperatures but for sufficient
time to produce very fine precipitates of average diameter less

35 than about 0.1 μm. After intermediate annealing ECAE is repeated

with the number of passes necessary to develop a dynamically
recrystallized structure with the desired fine and equiaxed
grains.

It is also possible through use of the invention to control
texture. Depending on the starting texture and the nature of the
materials, various textures can be created. Four major
parameters are important to obtain controlled textures:

Parameter 1: the number of repeated ECAE passes subjected
to the same work piece. This number determines the amount
of plastic deformation introduced at each pass. Varying
the tool angle between the two channels of the ECAE
equipment enables the amount of plastic straining to be
controlled and determined and therefore represents an
additional opportunity for producing specific textures.
Practically, in most cases, a tool angle of about 90° is
used since an optimal deformation (true shear strain ε =
1.17) can be attained;

Parameter 2: the ECAE deformation route; that is defined by
the way the work piece is introduced through the die at
each pass. Depending on the ECAE route only a selected
small number of shear planes and directions are acting at
each pass during plastic straining.

Parameter 3: annealing treatment that comprises heating the
work piece under different conditions of time and
temperature. Both post-deformation annealing at the end of
the ECAE extrusion and intermediate annealing between
selected ECAE passes are effective ways to create various
textures. Annealing causes the activation of different
metallurgical and physical mechanisms such as second-phase
particle growth and coalescence, recovery and static
recrystallization, which all affect more or less markedly
the microstructure and texture of materials. Annealing can
also create precipitates or at least change the number and

size of those already present in the material: this is an additional way to control textures.

Parameter 4: the original texture of the considered material.

Parameter 5: the number, size and overall distribution of second-phase particles present inside the material.

With consideration of these five major parameters, control of texture is possible in the ways described below:

Table 1 describes major components of texture between 1 and 8 ECAE passes via routes A through D in the as deformed condition for a strong initial texture and also for routes A and D for a weak initial texture. To describe major components both the 3 Euler angles ($\alpha\beta\gamma$) according to the Roe/Matthies convention and ideal representation {xyz} <uvw> are used. Moreover, the total volume percentage of the component is given. For texture strength both the OD index and Maximum of pole figures are given.

TABLE 1
Texture strength and orientation for route A
as a function of the number of passes and initial texture

Number of passes N	Major Texture Orientations *Notation:* Euler angles ($\alpha\beta\gamma$):{xyz}<uvw>:%total volume with 5° spread	OD index (t.r.)	Maximum of Pole Figures (t.r.)
	ROUTE A (STRONG INITIAL TEXTURE)		
Original (N=0)	(10.9 54.7 45):(-111)<1-23>:16% (105 26.5 0):(-102)<-28-1>:14% (110 24 26.5):(-215)<-5-5-1>:9.3%	21.7	17.02
N=1	(119 26.5 0):(-102)<-2-4-1>:17.62% (346 43.3 45):(-223)<2-12>:7.62%	10.9	10.9
N=2	(138 26.5 0):(-102)<-2-2-1>:8.66% (31 36.7 26.5):(-213)<-3-64>:8.6%	6.1	6.9
N=3	(126.7 26.5 0):(-102)<-2-3-1>:7.45% (21 36.7 26.5):(-213)<2-43>:6.1%	5.79	5.45

TABLE 1 (continued)
Texture strength and orientation for route A
as a function of the number of passes and initial texture

Number of passes N	Major Texture Orientations *Notation:* Euler angles (αβγ):{xyz}<uvw>:%total volume with 5° spread	OD index (t.r.)	Maximum of Pole Figures (t.r.)
N=4	(26.5 36.7 26.5):(-213)<2-64>:9.42% (138 26.5 0):(-102)<-2-2-1>:4.62% 169 15.8 45):(-115)<-32-1>:4.32%	4.82	6.55
N=6	(126.7 26.5 0):(-102)<-2-3-1>:6.66% (228 33.7 0):(-203)<-34-2>:5.8% (31 36.7 26.5):(-213)<3-64>:3.42%	3.94	5.61
N=8	(0 35.2 45):(-112)<1-11>:3.1% (180 19.4 45):(-114)<-22-1>:3.06% (31 25.2 45):(-113)<1-52>:2.2%	2.05	3.5
ROUTE A (WEAK INITIAL TEXTURE)			
Original (N=0)	(80 25.2 45):(-113)<8-11 1>:4.3% Large spreading around (106) (119)	2.6	3.2
N=1	(0 46.7 45):(-334)<2-23>:5.8% (222 26.5 0):(-102)<-22-1>:5% (128 18.4 0):(-103)<-3-4-1>:4.01%	4.02	6.3
N=2	(126.7 26.5 0):(-102)<-2-3-1>:6.22% (26.5 48.2 26.5):(-212)<1-22>:5.4% (162 13.2 45):(-116)<-42-1>:5.4%	4.4	6.8
N=4	(226 36.7 26.5):(-213)<-12-1>:4.85% (233 26.5 0):(-102)<-23-1>:4.63% (136 19.5 45):(-114)<-40-1>:4.54% (26.5 36.7 26.5):(-213)<2-64>:3.7%	3	5.1

TABLE 1 (continued)
Texture strength and orientation for route B
as a function of the number of passes and initial texture

ROUTE B (STRONG INITIAL TEXTURE)			
Number of passes N	**Major Texture Orientations** *Notation:* Euler angles $(\alpha\beta\gamma)$:{xyz}<uvw>:%total volume with 5° spread	**OD index (t.r.)**	**Maximum of Pole Figures (t.r.)**
Original (N=0)	(10.9 54.7 45):(-111)<1-23>:16.4% (105 26.5 0):(-102)<-2-8-1>:14% (110 24 26.5):(-215)<-5-5-1>:9.3%	21.7	17.02
N=1	(119 26.5 0):(-102)<-2-4-1>:17.62% (346 43 45):(-223)<2-12>:7.62%	10.9·	10.9
N=2	(0 48 26.5):(-212)<425>P24.24% (216 15.8 45):(-115)<-24-1>:8.07% (138 26.5 0):(-102)<-2-2-1>:5.04%	17.27	14.02
N=3	(260 36 74): (-2 7 10)<94-1>:15.49% (118 18.4 90):(013)<-63-1>:5.23%	7.3	9.1
N=4	(96 36 16):(-7 2 10)<-6-15-1>:12% (187 15.6 26.5):(-21-8)<-32-1>:8.05%	6	9.77
N=6	(230.5 14 0):(-104)<-45-1>:12.46% (100 36 16):(-7 2 10)<-4-9-1>:10.2%	6.3	8.45
N=8	(230.5 14 0):(-104)<-45-1>:9.19% (180 13.2 45):(-116)<-33-1>:8.21% (100 36 16):(-7 2 10)<-4-9-1>:7.48%	4.9	6.99

Table 1 (continued)
Texture strength and orientation for route C
as a function of the number of passes and initial texture

ROUTE C (STRONG INITIAL TEXTURE)			
Number of passes N	**Major Texture Orientations** *Notation:* Euler angles $(\alpha\beta\gamma)$:{xyz}<uvw>:%total volume with 5° spread	**OD index (t.r.)**	**Maximum of Pole Figures (t.r.)**
Original (N=0)	(10.9 54.7 45):(-111)<1-23>:16.4% (105 26.5 0):(-102)<-2-8-1>:14% (110 24 26.5):(-215)<-5-5-1>:9.3%	21.7	17.02
N=1	(119 26.5 0):(-102)<-2-4-1>:17.62% (346 43 45):(-223)<2-12>:7.62%	10.9	10.9
N=2	(0 34.5 14):(-416)<4-13>:43.3% (221.8 265 0):(-102)<-2 2 -1>:10.5%	48.9	25.9

TABLE 1 (continued)
Texture strength and orientation for route C
as a function of the number of passes and initial texture

	ROUTE C (STRONG INITIAL TEXTURE)		
Number of passes N	**Major Texture Orientations** *Notation:* Euler angles $(\alpha\beta\gamma)$:{xyz}<uvw>:%total volume with 5° spread	**OD index (t.r.)**	**Maximum of Pole Figures (t.r.)**
N=3	(254 148.4 0):(-103)<-3 11 -1>:7.5% (111.5 46.5 18.4):(-313)<-2-3-1>: 6.6%	5.2	6.05
N=4	(130 36.9 10):(-304)<-4-6-3>:15.05%	7.95	13.3
N=5	Large spreading (270 14 0):(-104)<010>:4.66% (26.5 48 26.5):(-212)<1-22>:2.54%	2.4	3.3
N=6	(110 36 16):(-7 2 10)<-5-8-2>:11.6% (234 33.7 0):(-203)<-35-2>:5.7%	6.32	9.3
N=7	Large spreading (242 18.4 0):(-103)<-36-1>:4.66% (188 11.4 45):(-117)<-34-1>:3.36%	2.35	3.15
N=8	(136.5 18.4 0):(-103)<-331>:14.71% (257 45 0):(-101)<-8 49 -8>:8.75%	12.9	11

TABLE 1 (continued)
Texture strength and orientation for route D
as a function of the number of passes and initial texture

Number of passes N	**Major Texture Orientations** *Notation:* Euler angles $(\alpha\beta\gamma)$:{xyz}<uvw>:%total volume with 5° spread	**OD index (t.r.)**	**Maximum of Pole Figures (t.r.)**
	ROUTE D (STRONG INITIAL TEXTURE)		
Original (N=0)	(10.9 54.7 45):(-111)<1-23>:16.4% (105 26.5 0):(-102)<-2-8-1>:14% (110 24 26.5):(-215)<-5-5-1>:9.3%	21.7	17.02
N=1	(119 26.5 0):(-102)<-2-4-1>:17.62% (346 43 45): (-223)<2-12>:7.62%	10.9	10.9
N=2	(0 48 26.5):(-212)<425>:24.24% (216 15.8 45):(-115)<-2 4 -1>:8.07% (138 26.5 0):(-102)<-2-2-1>:5.04%	17.27	14.02
N=3	(197 20.4 26.5):(-216)<-22-1>:9.57% all other components < 3%	3.91	6.67

TABLE 1 (continued)
Texture strength and orientation for route D
as a function of the number of passes and initial texture

	ROUTE D (STRONG INITIAL TEXTURE)		
Number of passes N	**Major Texture Orientations** *Notation:* Euler angles $(\alpha\beta\gamma)$:{xyz}<uvw>:%total volume with 5° spread	**OD index** (t.r.)	**Maximum of Pole Figures** (t.r.)
N=4	(222 26.5 0):(-102)<-22-1>:13.34% all other components < 3.8%	6.346	7.36
N=6	(223.5 18.5 0):(-103)<-33-1>:7.4% all other components < 2.5%	2.72	4.26
N=8	(222 26.5 0):(-102)<-22-1>:3.42% all other components < 3%	1.9	3.01
	ROUTE D (WEAK INITIAL TEXTURE)		
Original (N=0)	(80 25.2 45):(-113)<-8 -11 1>:4.3% Large spreading around (106) (119)	2.6	3.2
N=1	(0 46.7 45):(-334)<2-23>:5.8% (221 26.5 0):(-102)<-22-1>:5% (128 18.4 0):(-103)<-3-4-1>:4.01%	4.02	6.3
N=2	(241 26.5 0):(-102)<-24-1>:12.72% (26.5 48.2 26.5):(-212)<1-22>:4.1%	5	6.7
N=3	(197 20.~ 26.5):(-216)<-22-1>:8.8% (26.5 48.2 26.5):(-212)<1-22>:3.9%	3.5	6.44
N=4	(221.8 26.5 0):(-102)<-22-1>:7.2% (26.5 48.2 26.5):(-212)<1-22>:3.1%	3	5.3

Table 2 describes major components of features between 1 and 8 ECAE passes via route A through D for a strong initial texture and after annealing at (150C, 1h), (225C, 1h) and (300C, 1h)

Table 2

**Major texture orientations for route A
in function of number of passes N and annealing temperature**

ROUTE A (STRONG INITIAL TEXTURE)		
Notations: Euler angles (αβγ): {xyz} <uvw>: % total volume with 5° spread		
N	Annealing at (150C, 1h)	Annealing at (225C, 1h)	Annealing at (300C, 1h)
1	(43 47 22):(-525)<1-32>: 10.4% (110 26.5 0):(-102)<-2-6-1>:8.04% (130 24 18.4):(-317)<-3-2-1>:7.15%	(35 48 25):(-212)<1-22>:13.15% (114 22 10):(-102)<-2-4-1>:9.3%	(76 29.5 45):(-225)<-5-71>:9.3 % (141 37 0):(-304)<-4-4-3>:6.6 %
2	(105 22 0):(-205)<-5 20-2>:9.21% (155 19.5 45):(-114)<-31-1>:7.83% (31 36.7 45):(-213)<3-64>:6.88%	(136 18.4 0):(-103)<-3-3-1>:20.9% (112 19 18.4):(-319)<-5-6-1>: 20.2%	(354 18.4 0):(-103)<913>:7.74% (315 11.5 45):(-117)<701>:7.38% (90 7 0):(-108)<0-10>: 6.7%
3	(110 36 16):(-7 2 10)<-4-9-2>: 15.2% (233 26.5 0):(-102)<-23-1>:7.35%	(110 45 0):(-101)<-1-4-1>:16.85% (290 45 0):(-101)<141>:11.5%	Large spreading around (117),(100) All components < 4%
4	(129 18 26):(-217)<-1-5-1>:11.73% (35 37 26.5):(-213)<2-53>:11.2%	(124 25 14):(-419)<-3-3-1>:12.4% (38 36.7 26.5):(-213)<3-95>:7.5%	(110 25.2 45):(-113)<-6-3-1>: 6.87% (318 25.2 45):(-113)<301>:5.1%
6	(180 19.5 45):(-114)<-22-1>:5.5% (135 10 0):(-106)<-6-6-1>:4% (0 46.7 45):(-334)<2-23>: 3.95%	Large spreading All components < 5%	(46.7 19.5 45):(-114)<-1-17 4>: 9% All components < 4.9%
8	Large spreading around (315), (104) All components< 4%	(44 36 26.5):(-213)<2-63>:7.94% (136 18.4 0):(-103)<-3-3-1>:6.17%	(152 32 0):(-508)<-8-5-5>:6.4% All components < 3%

Table 2 (continued)
Major texture orientations for route B
in function of number of passes N and annealing temperature

ROUTE B (STRONG INITIAL TEXTURE)		
Notations: Euler angles (αβγ): {xyz} <uvw>: % total volume with 5° spread		
N	Annealing at (150C, 1h)	Annealing at (225C, 1h)	Annealing at (300C, 1h)
1	(43 47 22):(-525)<1-32>: 10.4% (110 26.5 0):(-102)<-2-6-1>:8.04% (130 24 18.4):(-317)<-3-2-1>:7.15%	(35 48 25):(-212)<1-22>: 13.15% (114 22 10):(-102)<-2-4-1>:9.3%	(76 29.5 45):(-225)<-5-71>:9.3 % (141 37 0):(-304)<-4-4-3>:6.6 %
2	(215 20 26.5):(-216)<-36 -2>:35% (270 13.2 45):(-116)<110>: 16%	(112 34 0):(-203)<-3-9-2>:16% (16 54.7 45):(-111)<1-34>:8.88%	(221 26.5 0):(-102)<-22-1>:13.3% (109 14 0):(-104)<-4-12-1>:12%
3	(148 19 79): (-1 5 15)<-55-2>:17.5% (90 16 45): (-115)<-1-10>: 6.9%	(10 45 10):(-616)<3-13>:5.7% (235 14 0):(-104)<46-1>:4.53% Large spreading	(0 48 26.5):(-212)<4-25>:6% (222 41 45):(-223)<03-2>:5.8% (19.5 45 0): (-101)<2-12>:5.4%
4	(127 26.5 0):(-102)<-2-3-1>:5.9% (242 14 0):(-104)<-4 8 -1>:	(230 14 0): (-104)<-45-1>:6.23%	Large spreading around (107) (115) All components < 3%
6	(180 19.5 45):(-114)<-22-1>:5.5% (135 10 0):(-106)<-6-6-1>:4 (0 46.7 45):(-334)<2-23>:3.95%	Large spreading All components < 5%	(46.7 19.5 45):(-114)<-1-17 4>:9% All components < 4.9%
8	Large spreading around (315), (104) All components<4%	(44 36 26.5):(-213)<2-63>:7.94% (136 18.4 0):(-103)<-3-3-1>:6.17%	(153 32 0):(-508)<-8-5-5>:6.4% All components<3%

Table 2 (continued)
Major texture orientations for route C
in function of number of passes N and annealing temperature

ROUTE C (STRONG INITIAL TEXTURE)		
Notations: Euler angles (αβγ): {xyz} <uvw>: % total volume with 5° spread		
N	**Annealing at (150C, 1h)**	**Annealing at (225C, 1h)**	**Annealing at (300C, 1h)**
1	(43 47 22):(-525)<1-32>: 10.4% (110 26.5 0):(-102)<-2-6-1>:8.04% (130 24 18.4):(-317)<-3-2-1>:7.15%	(35 48 25):(-212)<1-22>:13.15% (114 22 10):(-102)<-2-4-1>:9.3%	(76 29.5 45):(-225)<-5-71>:9.3 % (141 37 0):(-304)<-4-4-3>:6.6 %
2	(191 16 45):(-115)<-23-1>:8.77% (156 26.5 0):(-102)<-2-1-1>:6.68%	(99 46 14):(-414)<-3-8-1>:20.9% (289 45 0):(- 101)<141> :15.22%	Large spreading around (100) All components <3.8%
3	(119 26.5 0):(-102)<-2-4-1>:28.4% (26.5 48 26.5):(-212)<1-22>:9.74%	(106 29 26.5):(-214)<-5-6-1>:19.5% (103 31 34):(-326)<-6-6-1>:18.7% (42 46.5 18.4):(-313)<1-32>:8.83%	(194 14 0):(-104)<-41-1>:6.1% (163 18.4 0):(-103)<-3-1-1>:5.85%
4	105 38 18.5):(-314)<-3-5-1>:10.2% Other components <5.3%	Large spreading around (302) and (225) All components <2.8%	Large spreading around (100) (105) (116) All components <4.1%
5	(103 32 18.4):(-3 1 5)<-4-7-1>:19%	(127 26.5 0):(-102)<-2-3-1>:7% (22 38 18.4):(-314)<1-11>:5.6%	Large spreading around (106)(115) All components <3.7%
6	(61 46 14):(-414)<1-83>:11.82% (155 21 18.4):(-318)<-22-1>:7.94%	Large spreading around (101) and (334) All components <4%	(80 25 45):(-113)<-8-11 1>:4.3% All components <3%
7	(104 36 16):(-7 2 10)<-3-6-1>:29% (26.5 48 26.5):(-212)<1-22>:7.6%	(125 37 0):(-304)<-47-3>:7.8% (305 45 0):(-101)<121>: 5.82%	Large spreading around (100)(105)(203) All components <2.9%
8	(104 47 22):(-525)<-3-5-1>:15.36%	(106 38 18.4):(-314)<-3-5-1>4.64% All components <3.2%	Large spreading around (100)(105)(112)(203) All components <2.7%

Table 2 (continued)
Major texture orientations for route D
in function of number of passes N and annealing temperature

ROUTE D (STRONG INITIAL TEXTURE)		
Notations: Euler angles (αβγ): {xyz} <uvw>: % total volume with 5° spread		
N	Annealing at (150C, 1h)	Annealing at (225C, 1h)	Annealing at (300C, 1h)
1	(43 47 22):(-525)<1-32>: 10.4% (110 26.5 0):(-102)<-2-6-1>:8.04% (130 24 18.4):(-317)<-3-2-1>:7.15%	(35 48 25):(-212)<1-22>: 13.15% (114 22 10):(-102)<-2-4-1>:9.3%	(76 29.5 45):(-225)<-5-71>:9.3 % (141 37 0):(-304)<-4-4-3>:6.6 %
2	(215 21 26.5):(-216)<-36-2>:35% (270 13 45):(-116)<110>: 16%	(112 34 0):(-203)<-3-9-2>:16.45% (16 54.7 45):(-111)<1-34>:8.88%	(222 26.5 0):(-102)<-22-1>:13.3% (109 14 0):(-104)<-4-12-1>:12% (162 9 45):(-119)<-63-1>:9.6%
3	(337 50 34):(-323)<101>:12.2% (215 47 45):(-334)<0 4 -3>:9.75% (241 26.5 0):(-102)<-24-1>:7.02%	(168 20 25):(-216)<-82-3>:10.35% (102 18.4 0):(-103)<-3-16-1>:9.32% (162 13 45):(-116)<-42-1>:6.44%	(150 16 45):(-115)<115)<-41-1>:5.6% (198 18.4 0):(-103)<-31-1>:5.2%
4	(233 26.5 0):(-102)<-23-1>:9% All other components <4%	Large spreading All components <3.6%	Large spreading around (105) (116) All components < 3.9%
6	(224 18.4 0):(-103)<-33-1>:8.29% All other components <3.8%	(224 18.4 0):(-103)<-33-1>:5.49% (109 18.4 0):(-103)<-3-9-1>:4.4%	Large spreading around (106) and (113) All components < 2.9%
8	(222 27 0):(-102)<-22-1>:8.58% All components<4%	(205 21 18.4):(-138)<-22-1>:11.44% (233 26.5 0):(-102)<-23-1>:10.74%	(222 26.5 0):(-102)<-22-1>:8.58% (38 16 45):(-115)<1-92>:5.55%

(1)   The number of ECAE passes permits the control of texture strength.   The increase of the number of passes is an efficient mechanism of randomizing texture.   There is an overall decrease of texture strength evidenced by the creation

of new orientations and, more importantly, the large spreading
of orientations around the major components of the texture as
evidenced in FIG. 4. FIG. 4 is an illustration of (200) pole
figures for Al with 0.5 wt.% Cu alloys processed 2, 4 and 8
passes of route D (FIG. 5) and shows spreading of orientations
as "N" increases. This phenomenon is more or less effective
depending on the investigated route and/or annealing
treatment. For example in the as-deformed state, routes B and
C result in somewhat higher textures than routes A and D (FIG.
5 and Table 1). FIG. 5 is a graph that shows the influence of
ECAE deformation route and strength on texture formation as a
function of number of ECAE passes. For medium to very strong
starting textures, two main areas can be distinguished in the
as-deformed state(FIG.5):

Between passes 1 and 4 (with a tool angle of 90°), very
strong to medium textures are obtained. In the investigation
of Al.5Cu, for example, the OD index ranges from more than 7
times random to more than 48 times random which corresponds to
maximum intensities of the ODF between 3000 mrd (30 times
random) and more than 20000 mrd (200 times random).

For more than 4 passes (with a tool angle of 90°),
medium-strong to very weak textures close to random are
created. In the case of Al.5Cu alloys, OD index varies from
around 11 times random to less than 1.9 times random depending
on the route, which corresponds to maximum intensities of the
ODF between 7000 mrd (70 times random) and around 800 mrd (8
times random).

The two main domains are maintained after subsequent
annealing, as shown in the graphs of FIGS. 6, 7, 8 and 9.
However for some ECAE deformation routes (for example route B
and C in the case of Al.5Cu), additional heating can give a
strong texture, as discussed below. The existence of these
two areas is a direct consequence of the microstructural

changes occurring in the material during intensive plastic
deformation. Several types of defects (dislocations,

5   microbands, shear bands and cells and sub-grains inside these
shear bands) are gradually created during the 3 to 4 ECAE
passes (for a tool angle of 90°). The internal structure of
materials is divided into different shear bands while
increasing the number of passes. After 3 to 4 ECAE passes, a

10 mechanism termed dynamic recrystallization occurs and promotes
the creation of sub-micron grains in the structure. As the
number of passes increases these grains become more and more
equiaxed and their mutual local mis-orientations increase
giving rise to a higher number of high angle boundaries in the

15 structure. The very weak and close to random textures that
are created are a consequence of three major characteristics
of the dynamically recrystallized microstructures: the
presence of high internal stresses at the grain boundaries,
the large number of high angle boundaries and the very fine

20 grain size with a large grain boundary area (usually of the
order of about 0.1-0.5 μm).

    (2)   The ECAE deformation route permits control of the
major orientations of the texture. Depending on the route,
different shear planes and directions are involved at each

25 pass (see FIG. 5 and Tables 1 and 2). Therefore shear bands
of different orientations are created in the structure. For
some routes these shear bands always intersect each other in
the same way; for other routes new families are constantly
introduced at each pass (Tables 1 and 2). All these options

30 allow changes to the major components or orientations between
each pass. The effect is particularly strong for a small
number of passes before the advent of dynamic
recrystallization, as discussed above. An important
application exists in the possibility to create different

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types of strong textures already in the as-deformed state for a limited number of ECAE passes.

5   (3) Additional annealing has an important influence on both the major texture orientations and strength (see FIGS. 6, 7, 8, 9 and Table 2).

For annealing temperatures below the static recrystallization, a change in both texture strength and main

10 orientation is observed. This effect can be particularly strong for a low number of passes (less than about 4 passes) leading to remarkable migrations of major orientations accompanied with either a decrease or increase of texture strength. Such changes can be attributed to the instability

15 of microstructural defects which are implemented in the crystal structure. Complex mechanisms such as recovery and sub-grain coalescence explain partly the observed phenomena. For dynamically recrystallized ultra-fine structure (after usually 4 passes) smaller modifications are encountered. They

20 are usually associated with the transition from a highly stressed to a more equilibrium micro structure.

For annealing temperatures close to the beginning of static recrystallization, the same over-all results as in the above case are found. However, it is important to note that

25 new and different textures than for low temperature annealing can be obtained, especially for a low number of ECAE passes (Table 2). This is due to static recrystallization which creates new grains with new orientations by diffusion mechanisms.

30 For annealing temperatures corresponding to developed stages of static recrystallization (full static recrystallization), textures tend to be weakened (as shown in FIGS. 6, 7, 8, 9 and Table 2). This is particularly true after 3 or 4 ECAE passes where very weak and almost random

35 textures are created. These textures are characterized by

four, six or eight fold symmetry with a higher number of cube (<200>) components.

5   Additional textural analysis of ECAE deformed Al and 0.5 wt.% Cu is shown in the pole figure described in FIG. 10.   In this case the sample was given an initial thermochemical treatment of casting plus homogeneous plus hot forging plus cold rolling (~ 10%) plus two ECAE passes via route C plus

10   annealing (250°C, 1 hour).   The recrystallized microstructure had grain size of 40-60 μm and strong texture along {-111}<2-12>, {012}<-130>, {-133}<3-13>.   The result shows two ECAE passes (C) plus static recyrstalization permits removal of the very strong (220) textural component of the as-forged

15   condition.

        By taking into account all the foregoing, results show that intermediate annealing between each pass provides several additional and significant opportunities to adjust desired textures.   Two options are available:

20   A.   Intermediate annealing either at low temperature or
                just at the beginning of static recrystallization
                after a low number of passes (N<4) can give strong
                textures with new orientations after subsequent
                deformation with or without annealing.

25   B.   Intermediate annealing in the case of full
                static recrystallization after a low or
                high number of passes can lead more easily
                to very weak textures after subsequent
                deformation with or without annealing.

30   It is also possible to repeat intermediate annealing several times in order to enhance the effects described above.

        (4) Starting texture has also a strong influence on both texture and strength especially after a limited number of passes (usually after 1 to 4 passes).   For a higher number of

35   passes the ECAE deformation is very large and new mechanisms

are taking place which lessen the magnitude of the influence of the starting texture. Two situations are noted (FIG. 5 and

5   Table 1 for route A and D):

        A.   For a strong to medium starting textures, after further deformation with or without annealing, it is possible to obtain very strong to medium textures before 4 passes and strong-medium to very weak

10   textures after approximately 4 passes according to the results described in paragraph 1, 2 and 3.

        B.   For medium to very weak starting textures it will be more difficult to obtain very strong to strong textures at least in the as-deformed state. Weak

15   starting textures are more likely to enhance and promote weak to random textures after ECAE deformation with or without annealing (Table 1).

    (5)   Second phase particles have a pronounced effect on texture. Large (>1 μm) and non-uniformly distributed

20   particles are not desired because they generate many problems such as arcing during sputtering. Very fine (>1 μm) and uniformly distributed second phase particles are of particular interest and offer many advantages. Firstly, they tend to create a more even stress-strain state during ECAE

25   deformation. Secondly, they stabilize the already ECAE-deformed microstructure in particular after further annealing. In this case particles pin grain boundaries making them more difficult to change. These two major effects evidently affect the texture of materials. Especially:

30   for a small number of passes (<4 passes), the effects described previously in sections (1) to (4) can be enhanced due to the presence of second phase particles in particular for strong textures.

        for a large number of passes, second phase particles

35   are effective in promoting the randomization of texture.

In order to take advantage of the possibilities offered
by the ECAE technique in terms of texture control, three types

5   of results can be achieved:

A.   Materials (sputtering targets) with strong to very
strong (ODF>10000 mrd) textures. In particular this
can be obtained for a small number of passes with or
without subsequent annealing or intermediate

10   annealing. A strong starting texture is a factor
favoring the creation of strong textures. For
example in the case of A1.5Cu alloy Table 1 gives
all the major components of orientations which were
created for different deformation routes (A,B,C,D)

15   between 1 and 4 passes. The as-deformed state as
well as deformation followed either by low
temperature annealing (150°C, 1h) or by annealing at
the beginning of static recrystallization (225°C,
1h) or after full recrystallization (300°C, 1h) are

20   considered in this table. The original texture is
displayed in FIG. 7. It is important to note that
in most cases new types of textures have been found.
Not only {200} and {220} textures are present but
also {111}, {140}, {120}, {130}, {123}, {133}, {252}

25   or, for example, {146}. For strong textures, one or
two main components are usually present.

B.   Material (sputtering targets) with weak to close to
random textures with an ultra-fine grain size less
than 1μm. Whatever the route this can be obtained

30   after more than 3 to 4 ECAE passes followed or not
by annealing or intermediate annealing at a
temperature below the beginning of recrystallization
temperature. A very weak starting texture is a
factor favoring the creation of close to random

35   textures.

-25-

C.   Statically recrystallized materials (sputtering
      targets) with weak to close to random textures with
      a fine grain size above approximately 1 μm.
      Whatever the route this can be obtained after more
      than 3 to 4 ECAE passes followed by annealing or
      intermediate annealing at a temperature above the
      beginning of recrystallization temperature. A very
      weak starting texture is a factor favoring the
      creation of close to random textures.

Another embodiment of the invention is an apparatus for
performing the process to produce targets. The apparatus
(FIGS. 11, 11A and 11B) includes die assembly 1, die base 2,
slider 3, punch assembly 4,6 hydraulic cylinder 5, sensor 7,
and guide pins 11. Also the die is provided with heating
elements 12. Die assembly 1 has a vertical channel 8. A
horizontal channel 9 is formed between die assembly 1 and
slider 3. The die is fixed at table 10 of press, punch
assembly 4, 6 is attached to press ram. In the original
position a-a the forward end of slider 3 overlaps channel 1,
punch 4 is in a top position, and a well lubricated billet is
inserted into the vertical channel. During a working stroke
punch 4 moves down, enters channel 8, touches the billet and
extrudes it into channel 9. Slider 3 moves together with
billet. At the end of stroke the punch reaches the top edge
of channel 9 and then returns to the original position.
Cylinder 5 moves the slider to position b-b, releases the
billet, returns the slider to the position a-a and ejects the
processed billet from the die. The following features are
noted:

      (a) During extrusion slider 3 is moved by hydraulic
cylinder 5 with the same speed as extruded material inside
channel 9. To control speed, the slider is provided with
sensor 7. That results in full elimination of friction and

material sticking to the slider, in lower press load and
effective ECAE;

5   (b)   Die assembly 1 is attached to die base 2 by guide
pins 11 which provide free run δ. During extrusion the die
assembly is nestled to the base plate 2 by friction acted
inside channel 8. When the punch returns to the original
position, no force acts on the die assembly and slider, and
10 cylinder 3 can easily move the slider to position b-b and then
eject the billet from the die.

        (c)   Three billet walls in the second channel are formed
by the slider (FIG. 11A) that minimizes friction in the second
channel.

15   (d)   The side walls of the second channel in the slider
are provided with drafts from 5° to 12°. In this way the
billet is kept inside the slider during extrusion but may be
ejected from the slider after completing extrusion. Also,
thin flash formed in clearances between the slider and die
20 assembly may be easily trimmed.

        (e)   Die assembly is provided with heater 12 and springs
13. Before processing, springs 13 guarantee the clearance δ
between die assembly 1 and die base 2. During heating this
clearance provides thermoisolation between die assembly and
25 die base that results in short heating time, low heating power
and high heating temperature.

        The apparatus is relatively simple, reliable and may be
used with ordinary presses.

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